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### VAPOR PHASE GROWTH OF RUBY MONOCRYSTALS

#### Final Technical Summary Report

1 May 1964 - 31 August 1967

Robert C. Folweiler - Author

Contract No. Nonr-4574(00)-2  
ARPA Order - 306

December 1967

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## FOREWORD

This report was prepared by Robert C. Folweiler of Lexington Laboratories, Inc., 84 Sherman Street, Cambridge, Massachusetts 02140, under Contract Nonr-4574(00)-2, covering the period July 1966 to July 1967. Contract monitor was Dr. Van O. Nicolai of the Office of Naval Research.

P. S. Schaffer was principal investigator from the inception through April 1967, when R. C. Folweiler assumed the duties. R. C. Folweiler and H. A. Hobbs designed the furnace configuration and accessory equipment. Outstanding support was provided by L. G. Terrenzio and B. A. Albanese throughout. Many helpful suggestions and comments were provided by R. L. Coble and W. D. Kingery.

## ABSTRACT

Improvements in apparatus and control of process parameters have led to the growth of a ruby weighing more than 200 grams. Several major limiting features of the process including furnace size, crystal purity, doping techniques, and pumping systems were improved in conjunction with a continuing study of process parameters. A significant breakthrough was made when a furnace construction technique was developed which did not limit furnace size. A four inch inside diameter furnace was constructed using this technique and has performed satisfactorily. In an investigation of doping techniques, additional sources of chromium vapor have been investigated, and chromyl chloride ( $\text{CrO}_2\text{Cl}_2$ ) has proved to be satisfactory.

Previous measurements of laser characteristics determined have demonstrated that vapor grown ruby is of high optical quality and compares favorably to other available crystals in pumping behavior, light scattering, and interferometric properties.

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## 1.0 INTRODUCTION

Single crystals of aluminum oxide in both doped and undoped forms have been synthesized for a number of years utilizing the Verneuil technique, and in the past decade the Czochralski technique has been applied to production of high quality ruby and sapphire. Almost no effort, however, was expended on utilization of the chemical vapor deposition process for synthesis of useful single crystal material of aluminum oxide until about five years ago.

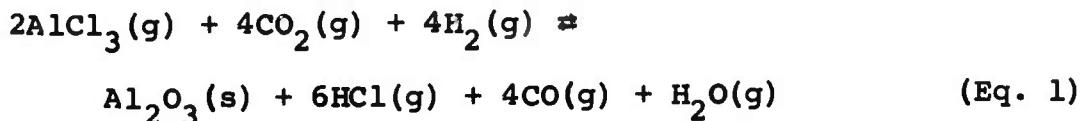
Single crystals of other materials have been synthesized by vapor transport and/or reaction techniques, but for the most part, the results have been either small crystals or films<sup>1</sup>. For instance, considerable effort has been expended in attempting chemical vapor deposition of silicon carbide with the continuing result that little progress has been made since most crystals are of millimeter size<sup>2</sup>. The most significant use of chemical vapor deposition techniques at the present time has been for the production of both highly-oriented and isotropic polycrystalline materials such as pyrolytic graphite, boron nitride, and a variety of refractory metallic and intermetallic compounds. Although these materials have not enjoyed great economic success, the chemical vapor deposition process continues to hold considerable promise for certain geometric forms, and specific materials which are best produced by this process. The process can take place isothermally with no thermal strain resulting from the growth process. In addition, purity may be improved substantially by the application of various techniques which can be utilized only in this process. Finally, a number of adjustable variables are available by which one may change the stoichiometry and properties of the deposited material.

## 2.0 REVIEW OF PREVIOUS WORK

Previous work done under this contract is summarized in this section<sup>3,4,5</sup>.

### 2.1 Overall Process

The basic process utilizes the controlled oxidation of a volatile aluminum compound, aluminum chloride ( $AlCl_3$ ). The source of oxygen used has been carbon dioxide ( $CO_2$ ), which at the temperatures involved dissociates:  $CO_2 \approx CO + 1/2O_2$ . The process proceeds utilizing the following overall reaction:



Aluminum chloride is generated by direct chlorination of aluminum metal because of the relative ease with which chlorine gas may be metered directly using a standard flow meter compared to either the direct monitoring of aluminum chloride flows (which would require more difficult metering techniques) or indirect techniques such as evaporation and transport by a carrier gas. High purity aluminum metal as a source material is readily available, as is chlorine of reasonable purity.

A stoichiometric excess of hydrogen is used to reduce one of the thermodynamic variables. Addition of one of the product gases, CO, improves the stability of the growth process, probably due to the reduction of the initial thermodynamic driving force.

### 2.2 Doping Techniques

Doping of the product with chromium in order to produce ruby required replacing part of the aluminum in the oxide lattice by chromium. While perhaps superficially simple, the substitution was difficult because of subtle

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very high aluminum with small amount of aluminum in the  
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the strength was quite different. It might seem that the  
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### 2.3 Purity

The nature of the process leads to an overall purification which decreases the impurities which may be carried into the reaction chamber. Differential chlorination and evaporation rates in the chlorinator, plus different deposition coefficients in the reaction chamber lead to enhanced purity. Qualitative spectrochemical analyses indicated typical impurity contents in the range 10 - 30 ppm and as low as <3 ppm cation impurities. No highly quantitative determinations have been made for all the cation impurities.

### 2.4 Crystalline Perfection

Dislocation density observed in a vapor grown ruby crystal, reported previously, were observed to be  $6.2 \times 10^{-5}$  per  $\text{cm}^2$  on the prism plane  $\{11\bar{2}0\}$ , a substantial improvement over the flame-fusion material used for the substrate. Undoped sapphire demonstrated a lower dislocation content, and a vapor nucleated and grown crystal demonstrated a dislocation density of 13 per  $\text{cm}^2$ .

### 2.5 Laser Properties

A vapor grown rod doped with approximately 0.1 wt % chromium was tested for laser properties<sup>8</sup>. Pumping energy requirements were observed which were similar to ruby prepared by other techniques.

### 3.0 EXPERIMENTAL APPARATUS

The emphasis of the recent contractual period included major improvement in two areas: (1) size of final crystal and (2) doping techniques. Experiments were performed which led to substantial redesign of our reaction chambers.

#### 3.1 Furnace Configuration

The most important change developed for the furnace configuration was the removal of the inherent size limitation placed on the reaction chamber resulting from use of impervious aluminum oxide tubes. These tubes, which contained the growth process at operating temperature and isolated it from the ambient atmospheric pressure, were a substantial source of difficulty. Frequent internal defects, plus geometric defects such as eccentricity led to failures of the tube under operating conditions. This problem became a serious difficulty as the size of the tube was increased over two inches inside diameter. Experiments indicated that molybdenum was stable in the slightly oxidizing atmosphere used for the reaction; it was therefore concluded that in all likelihood, a molybdenum heating element could be operated within the growth atmosphere without deleterious effects.

A furnace was designed and constructed based on this observation; its structural arrangement is indicated in Figure 1. A steel shell isolated the reactor from the external atmospheric pressure. A molybdenum heating element was helically wound on a porous aluminum oxide tube which provided mechanical support for the winding. A variety of insulating materials, powders, foams, and bubbles, were successfully utilized to provide thermal insulation. Flanges

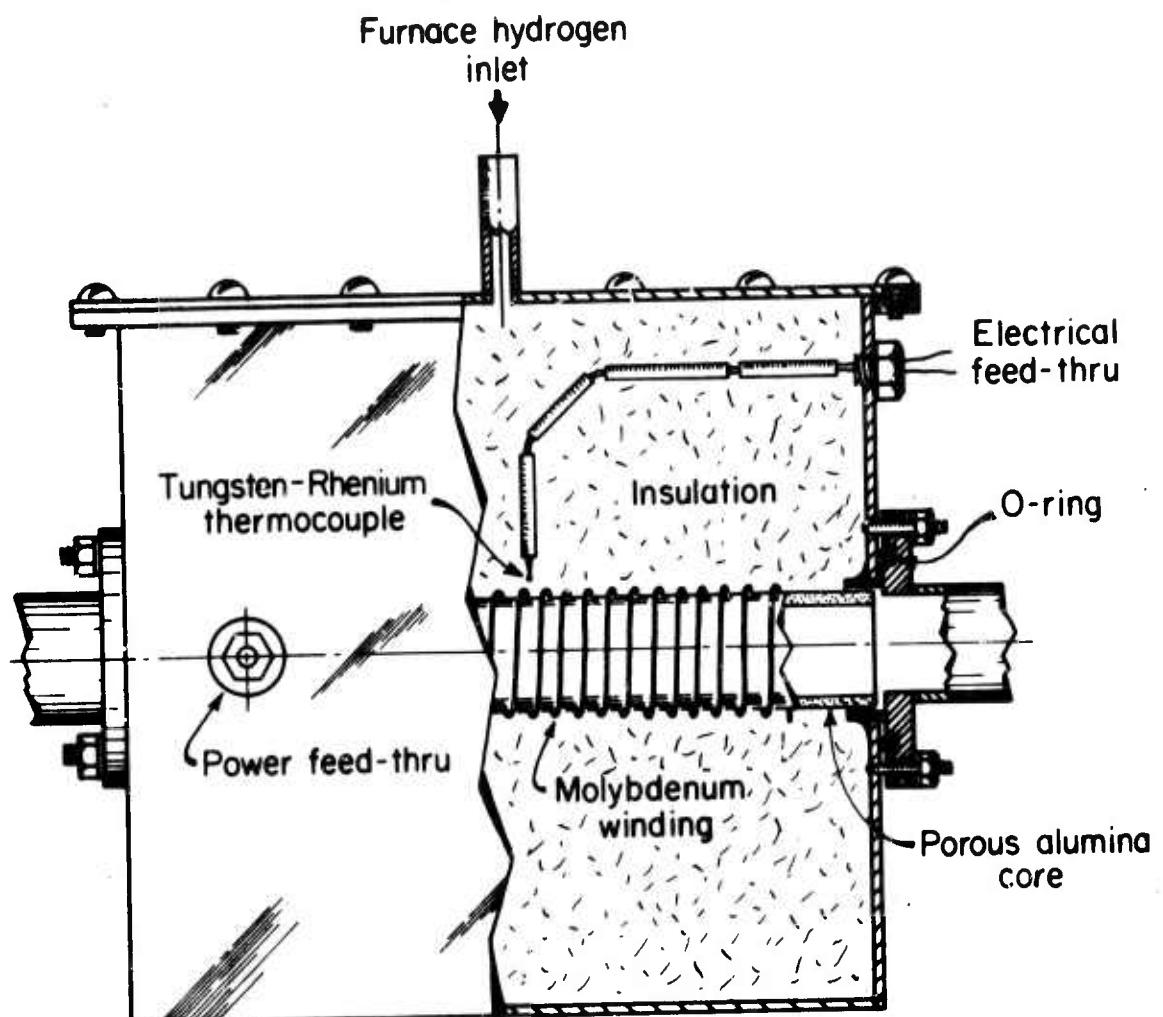


Figure 1: Schematic of Furnace Design.

were provided for the injector and for the exhaust system.

The initial power supply for the larger inside diameter furnace utilized a silicon controlled rectifier (SCR) unit with the control signal obtained by monitoring the resistance of the winding element. It was subsequently found that at low pressures (under three Torr) electrical breakdown occurred within the furnace leading to failure of the SCRs or protective fuses, since they could not tolerate unusually high peak currents. This difficulty was alleviated in two different ways: 1) the pressure was maintained above this critical pressure and 2) the power supply was changed to a variable autotransformer type, as the latter can tolerate substantial short-term peak currents. Within the appropriate restrictions, both power supplies operated satisfactorily.

The utility of a tungsten-rhenium alloy thermocouple with the hot junction adjacent to the winding was investigated. The thermocouple proved generally adequate although failures were experienced when a rapid shut-down occurred, requiring the replacement of the thermocouple.

### 3.2 Furnace Safety Apparatus

Because the larger system involved substantially larger volumes of hydrogen than previously, and because there was a danger that the molybdenum winding might suffer catastrophic oxidation, a safety system was designed to operate upon signal from an aneroid pressure switch. Upon activation of this switch, a relay a) shut down the pumps, b) shut off the reactant gases, c) flooded the furnace with a non-flammable quenching gas at a high volume rate and d) turned off the power supply. The non-flammable quenching gas (90% helium, 10% hydrogen) chosen provided adequate

stability for the winding while simultaneously protecting personnel. The system has pressurized the furnace to atmospheric within two seconds and provided a small positive pressure in order to maintain outward diffusion.

### 3.3 Vapor Train Modifications

Previous work indicated substantial difficulty with the chlorine vapor train. Stainless steel tubing was not adequate, since substantial corrosion occurred on the interior surface. Most of the transfer system was replaced with polymers (polyethylene, polyvinyl chloride, polypropylene and tetrafluoroethylene). With the exception of some minor stress cracking in the polypropylene fittings, these materials performed well. The vapor transfer line for the aluminum chloride were changed to aluminum oxide tubing.

An attempt was made to eliminate the corrosion of certain stainless steel components (valves, meter tube fittings and related parts) by coating exposed surfaces with fluorinated ethylene-propylene (FEP). The coating was permeable to chlorine and did not eliminate the reaction.

### 3.4 Doping Techniques

Doping was previously performed by the direct chlorination of chromium metal in a boat adjacent to the growth area. Difficulties lead to the study of several other potential doping techniques including chromium carbonyl, chromium acetyl acetonate and chromyl chloride. These compounds have a substantial vapor pressure at temperatures near room temperature and thus may be handled in exterior generators.

The carbonyl and the acetyl acetonate were transported through the use of a carrier gas. Hydrogen,

carbon monoxide, and carbon dioxide were used. Both of the chromium compounds were vaporized from the solid state by heating them in a well stirred bath. Chromyl chloride ( $\text{CrO}_2\text{Cl}_2$ ) is a liquid with a vapor pressure of approximately 20 Torr at 20°C. It was introduced into the system by utilizing chlorine as a carrier gas, saturating the chlorine with the compound by passing it through a gas washer in a controlled temperature bath.

### 3.5 Downstream Modifications

In earlier furnaces we had difficulty with both the pumping apparatus and the cooler sections of the furnace. Powder depositing from the flow stream clogged the exit tube after extended periods of operation. Provisions were made for a scraping device to remove any substantial build-up of powder before excessive difficulty occurred with the reaction.

We continued to utilize oil sealed mechanical pumps with a liquid nitrogen trap ahead of the pumps. In order to handle the increased capacity requirements for larger furnaces, pumps were manifolded and the liquid nitrogen trap enlarged. Total pressure control inside the reaction chamber was performed by a nitrogen ballast which loaded the pumps to capacity at the desired pressure.

### 3.6 Seed Orientation

Previous difficulties with the axial variation of chromium homogeneity along the rod led to a change in mounting orientation. The seeds in the new larger furnace were mounted in a "cross flow" configuration, whereby the long axis of the seed was held vertical, perpendicular to the flow stream. An aluminum oxide plaque was used to support the seeds.

This technique was considered advantageous because the seed lay in a single cross section within the furnace, where the chromium distribution was expected to be uniform. It also removed a downstream "seed" support assembly which had been used and caused difficulties both from creep-deformation during operation and from blockage due to interference with the flow stream.

### 3.7 Injector Design

Previous difficulties with injector plugging led to a redesign of that assembly. The new design consisted of two concentric tubes and a third smaller tube. During sapphire growth, the smaller tube introduced the aluminum trichloride into the outer annulus between the furnace wall and the large injector tube. The inner annulus conducted the reducing gases, CO and H<sub>2</sub>. The innermost tube carried the oxidizing gas, CO<sub>2</sub>, plus Cl<sub>2</sub> when utilized. All tubes ended at approximately the same distance into the furnace. The intent was to use the reducing gases to form a separating wall between the AlCl<sub>3</sub> and the CO<sub>2</sub> until the gases had moved downstream from the injector, thus avoiding reaction in the immediate area of the injector. This separation led to substantially reduced growth on the injector when growing sapphire.

When CrO<sub>2</sub>Cl<sub>2</sub> was used as a dopant medium during ruby growth, however, we found that the presence of the CO<sub>2</sub> in the vapor train with the CrO<sub>2</sub>Cl<sub>2</sub> in the inner tube caused premature reaction and resultant plugging. While growing ruby therefore, we have moved the CO<sub>2</sub> so that it is introduced with the CO and H<sub>2</sub>. This change resulted in slightly more growth on the injector system than noted during sapphire runs.

## 4.0 RESULTS AND DISCUSSION

The furnace system described in the preceding section has been operated for substantial periods of time. This improved apparatus, plus a better understanding of process variables has led to a substantially increased crystal size. A number of process variables and operating techniques were changed or adjusted until satisfactory growth was obtained.

### 4.1 Furnace Assembly

The modified furnace assembly with its newly designed pressure vessel, vapor train, and pumping modifications was continuously operated for a period exceeding 50 hours. A number of operational difficulties occurred, which led to apparatus modifications. Most were minor and readily corrected, except the problem of internal electrical breakdown. As mentioned previously, the problem of breakdown was controlled by the maintenance of an appropriate total pressure within the furnace.

### 4.2 Crystal Growth

Several experimental crystal growth runs were conducted utilizing the cross-flow arrangement. The results of one of the runs are shown in Figure 2, indicating one of the problems of this configuration. The resultant crystals had re-entrant surfaces and were substantially barrel-shaped, which is considered an unsatisfactory configuration for the final product. This barrel shape can be directly ascribed to the laminar flow conditions within the furnace. Since the flow was laminar ( $N_{Re} \sim 1$ ), the velocity profile across the tube was approximately parabolic, which demonstrates that the flow velocity strongly influences growth rate. It



Figure 2: Rubies Grown Simultaneously in Horizontal "Cross Flow" Furnace.

was also apparent, even though  $N_{Re}$  was small, that there were turbulent eddies which occurred after a period of growth leading to the distorted forms as seen in the cross section.

In addition, substantial chromium banding within the crystals occurred which is visible in the inset of the photograph. This chromium banding was due to two factors, one of which was difficult to control. The banding was due in part to inadequate controls of the chromium generation apparatus, whether it be direct chlorination of the chromium metal or the introduction of a chromium compound. It seems fairly certain that the input flow variations which cause banding can be controlled adequately by appropriate commercially available methods. The problem of local eddies adjacent to the crystal is a substantially more difficult problem and would require modifications of the furnace assembly to permit rotation of the crystal. Preliminary studies have indicated that this would not be difficult to accomplish.

It is not clear at this time, however, that the cross-flow configuration is advantageous for growing rod-shaped crystals. A rectangular reaction chamber cross section, rather than the present circular cross section, would be required to avoid the barrel-shaped effect. By the use of this modified reaction chamber cross section, it is anticipated that large size cross-flow rubies could be grown.

An axial flow ruby was grown to substantial size as shown in Figure 3, in a furnace with vertical flow orientation<sup>9</sup>. This ruby retained the usual axial variation in chromium content previously experienced. However, the



Figure 3: Largest Ruby Grown by Chemical Vapor Deposition.

chromium variation was substantially lower than we have previously encountered and indicated that we can substantially improve the axial chromium variation by changes in the input gas flows. Almost all of the radial banding can be directly attributed to external variations in the gas saturation apparatus, either temperature or pressure. Steps have been taken to improve the control of these variables and this problem has been largely solved, although further experimentation is essential to determine the degree of control required.

The addition of the doping apparatus to the growth system required a shorter injector assembly in order to reduce the temperature at the exit of the injectors. This was done in an effort to reduce undesired pre-reaction in the general area of the end of the injector. Because the growth pattern of ruby was different from that observed on a sapphire run of otherwise similar parameters, it was concluded that a higher gas velocity is required in the furnace during ruby growth in order to produce a more desirable growth configuration. However, with the present pumping equipment, adequate velocity is not attainable.

Satisfactory seed material continues to be a substantial problem. Available Verneuil material is of low quality and, along with Czrochalski material, has an inappropriate orientation for the most favorable vapor growth. A low angle orientation, preferably with the "c"-axis co-axial with the rod, has the best overall growth characteristics. Because of the epitaxial growth process, substrate misorientation defects continue into the overgrowth.

#### 4.3 Laser Evaluation

Banding and internal defects were sufficiently extensive that evaluation of laser properties was not

considered desirable in the crystals grown under this portion of the contract.

#### 4.4 Crystalline Purity

A sample of vapor grown material was analyzed by nuclear activation techniques for chlorine content. The analysis indicated a concentration of between four and six parts per million (PPM) chlorine<sup>10</sup>, significantly lower than the sample previously analyzed by vacuum fusion and reported to contain 0.15 wt. % chlorine<sup>5</sup>.

## 5.0 SUMMARY

5.1 Significantly larger ruby crystals (over 200 grams) were grown utilizing the chemical vapor deposition process than previously accomplished.

5.2 A new furnace construction technique was developed and proved which does not place any basic limit on crystal size.

5.3 New sources of chromium dopant were investigated. Chromyl chloride demonstrated some advantages over the direct chlorination of chromium metal.

5.4 Procurement of satisfactory substrate material presents a limit on the growth of quality material.

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13. ABSTRACT
Improvements in apparatus and control of process parameters have led to the growth of a ruby weighing more than 200 grams. Several major limiting features of the process including furnace size, crystal purity, doping techniques, and pumping systems improved in conjunction with a continuing study of process parameters. A significant breakthrough was made when a furnace construction technique was developed which did not limit furnace size. A four inch inside diameter furnace was constructed using this technique and has performed satisfactorily. In an investigation of doping techniques, additional sources of chromium vapor have been investigated, and chromyl chloride ( $\text{CrO}_2\text{Cl}_2$ ) has proved to be satisfactory.

Previous measurements of laser characteristics determined have demonstrated that vapor grown ruby is of high optical quality and compares favorably to other available crystals in pumping behavior, light scattering, and interferometric properties. (DLC)

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